WATER RESOURCES RESEARCH GRANT PROPOSAL

Title: ***Occurrence of Nonylphenolethoxylate (NPE) Surfactants and their Metabolites in New Jersey Rivers and Wastewater Treatment Plants***

Investigator:

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Amount Requested: $25,000.00

Overview

Surfactants have been shown to be very effective in soil-washing, flushing technologies and bioremediation of contaminated sites. The improved effectiveness of these surfactant-amended systems is due to the solubilization of adsorbed hydrophobic compounds in soils. They have been shown to increase solubility and dispersion of poorly soluble hydrocarbons and oils thereby enhancing desorption and bioavailability. The use of surfactants to enhance the aqueous solubilization of hydrocarbons (such as, Non-aqueous phase liquid-NAPLs and polycyclic aromatic hydrocarbons etc.) has been investigated extensively in recent years by a number of investigators (Ellis et al., 1987; Vigon and Rubin, 1989; Kile and Chiou, 1989; Liu et al., 1991; Sun and Boyd, 1995; Butler and Hayes, 1998). Researchers have indicated that solubilization followed by microbial metabolism of organic contaminants is technically feasible and has potential as a remedial technology (Pritchard et al., 1994). Addition of surfactants, detergents and emulsifiers has been successfully applied for cleanup of petroleum contaminated sites (Ellis et al., 1987). However, recently a certain group of widely used alkylphenolethoxylates (APEs) surfactants were banned in Europe because scientists discovered that APE breakdown products are highly toxic to aquatic organisms. Recent evidence that some APE breakdown products are **estrogenic** has intensified concern over their environmental and human health effects (Renner, 1997). Throughout northern Europe a voluntary ban on APE use in household cleaning products began in 1995 and restrictions on industrial applications are set to follow by the year 2000. The City of Toronto in Canada has also banned discharges of APEs into their sewer systems.

APEs are nonionic surfactants made up of a branched chain ethylene oxide to produce an ethoxylate chain. The main alkylphenols used are nonylphenol (NP) and octylphenol (OP). Nonylphenol ethoxylates (NPEs) encompass about 80% of the world market, and octylphenol ethoxylates (OPEs) represent most of the rest (Warhurst, 1995). They have been used extensively for their effectiveness, economy and ease of handling and formulating for more than forty years. They function as emulsifiers, wetting agents and dispersants. The primary industrial uses of APEs are for emulsion polymerization and polymer stabilization in plastics and elastomers; cleaning, spinning, weaving, and finishing of textiles; wetting agents and emulsifiers in agricultural chemicals; and pulping and deinking in the paper industry. Institutional use of APEs are confined to cleaning products, and most are found in commercial laundry detergents, janitorial products, and vehicle cleaners. In the household market, APEs are used mainly in laundry detergents and hard-surface cleaners. *Most APEs enter the aquatic environment from wastewater treatment plant discharges.*

APE biodegradation accomplished by stepwise shortening of the ethoxylate chain creates a complex mixture of compounds that can be divided into three main groups: short-chain ethoxylates, alkylphenoxy carboxylic acids, and alkylphenols such as NP and OP. As the chain becomes shorter, the molecule becomes less soluble. The alkylphenoxy carboxylic acids and longer chain APEs are soluble in water; the shorter chain APEs are insoluble in water; particularly NP and OP, have low water solubility and tend to adsorb onto suspended solids or sediments. Most studies and regulations focus on NPEs, because these are the most widely used. Nonylphenol (NP), one of the breakdown products, is also approximately 10 times more toxic than its ethoxylates. The generic NPE structure is depicted in Figure 1. The average number of moles of ethylene oxide per mole of NP (n) ranges from 1 to 100.

C_9H_{19}- -O $(CH_2CH_2O)_n$ H (n=number of ethylene oxides; range 1-100)

Figure 1: Nonylphenol ethoxylate structure

Relevance to Needs and Research Plan

In the United States, industrial uses of APE encompass the largest category (55%). *There are no U.S. regulatory actions to date for APEs*. U.S. and European regulators and researchers disagree strongly over the risks of APEs. European studies (Giger et al., 1984; Ahel et al.,1994) indicate that many sewage treatment works discharge substantial amounts of APE metabolites. Very few detailed studies have been conducted in the U.S. on the toxicity of APEs and their presence in wastewaters. Field and Reed (1996) conducted tests on 15 paper mills and 6 publicly owned treatment works (POTWs) discharging to Wisconsin’s Fox River. Results indicated that APEs are a problem on the Fox river. The U.S. producers of NP and NPEs in 1987 formed a panel within the CHEMSTAR Division of the Chemical Manufacturers Association (Naylor et al., 1992; Kubeck and Naylor, 1990). The mission of the alkylphenol and ethoxylates panel was to critically evaluate the published information on the environmental fate, degradaton and toxicity of NPEs. Major accomplishments of the panel include:

· Negotiation of a consent order with EPA under TSCA Section 4

· Completion of all environmental effect tests required under the consent order

· A voluntary limited river monitoring study, designed with assistance from the EPA

· Development and validation of analytical methods capable of measuring NP and NPEs at the sub- part-per-billion level

· Treatability evaluation on NPEs on a variety of wastewater treatment plants

This work was funded by six major chemical companies (Huntsman, Union Carbide, Rhone-Polenc, General Electeric, Witco and Exxon Chemical). The major conclusions of this study were:

· **NPEs are highly biodegradable in the environment,**

· **NPEs do not accumulate in water, sediment or aquatic organisms and**

· **NPEs do not pose a credible threat to the environment.**

This conclusion of course is totally the opposite of that determined by European researchers. One of the interesting results of this study were the two New Jersey rivers, Egg Harbor and Delaware indicating high exposure ratings for NP and NPEs out of the total of 30 rivers studied in the USA. None of the wastewater treatment plants studied were located in New Jersey. However, most wastewater treatment plants in New Jersey receive APE contaminated wastewaters from chemical industries (e.g., Rahway Valley Sewerage Authority) and are facing operational problems.

Therefore, more research on the fate and transport of APEs in the environment is essential. This study attempts to characterize the occurences of APEs in the river waters, sediments and wastewater treatment plants of New Jersey. This will be an extremely valuable supplemental study as the state of New Jersey has a strong industrial base comprising of pharmauceuticals, chemical manufacturers and food industries. All these industries are major users of this class of surfactants that are undergoing bans in Europe. Two letters of support have been included from local wastewater utilities.

Relevant Research Conducted by the PI

This proposal aims at studying the fate and transport of common commercial nonionic surfactants specifically the NPE’s in New Jersey rivers and wastewater treatment plants. Experiments to identify biodegradation intermediates will be conducted and toxicity tests will also be performed on the intermediates.

The PI has extensive research experience with surfactant enhanced bioremediation of hydrophobic compounds. Her doctoral research focussed on the effect of nonionic surfactants in enhancing the solubility and biodegradation rates of phenanthrene, a PAH compound. Mathematical modeling of experimental data to elucidate the mechanisms involved in surfactant enhanced remediation of PAHs was also an integral part of the study. Her research results have led to 12 technical publications and a number of presentations.

At Rowan, the PI has conducted preliminary investigation on the biodegradability of nonionic surfactants when she received the Lindback Foundation's Junior Faculty Award in 1997 and an undergraduate student stipend from the NJWRRI. Acclimated cultures were developed from mixed liquor collected from the local wastewater treatment plant. Batch biodegradation studies were conducted in the HACH BOD Trak apparatus with PC based data acquisition system. Two sophomore undergraduate students were involved with the following experiments:

· development of enrichment cultures,

· characterizing sorption of nonionic surfactants to a low organic carbon content sand (Penn sand), and

· characterizing biodegradability of nonionic APE surfactants.

To date, tests have been conducted to assess the biodegradability of two APE surfactants: Tergitol NP-10 and Triton X-114 (Sigma Chemicals, St. Louis, Mo). Tergitol NP-10 is a nonylphenol while Triton X-114 is an octylphenol ethoxylate. Surfactant concentrations were measured spectrophotometrically (Model DR2000, HACH Co., Loveland, CO.) and oxygen uptake rate was tracked by the HACH BOD Trak apparatus. Experiments were conducted at 20^{o}C in an incubator. About 60% of the Tergitol surfactant was removed in about 18 days. *Biodegradation intermediates of the surfactants were not identified in these experiments. The fate of the intermediates were also not studied*. Batch sorption experiments were also conducted to characterize the sorptive properties of these surfactants to a sand with a very low organic carbon content. Results of this research were published and presented at last year's annual conference of the Water Environment Federation (Jahan et al., 1998). Undergradauate students involved in this research won the best paper award at last year's National Engineers' week sponsored by the Delaware Valley Engineers Council and also won the first prize in a poster competition arranged by the New Jersey Water Environment Association.

The PI was contacted by the Rahway Valley Sewerage Authority to study the occurrence of nonionic surfactants and their effect on foaming in their wastewater treatment plant.

Nonionic surfactant concentrationsas CTAS (Standard Methods, 1998) were monitored at various locations of the plant. Foaming potential of commercial surfactants were also evaluated using the method outlined by Ho and Jenkins (1997). Results from this study indicated that the plant only removes 70% of the surfactants. The removal efficiency is much lower than that reported by European researchers (Giger, 1988). Research results were presented at this year's Annual Water Environment Federation Conference at New Orleans, Louisiana (Jahan et. al., 1999). *The sludge at the RVSA facility was not monitored for residual surfactant concentrations.*

The PI has been awarded a grant from the Northeast Hazarodous Substance Research Center for conducting laboratory experiments on the fate and transport of nonionic surfactants. Experiments are in progress to assess the biodegradability of nonylphenol using acclimated cultures from wstewater. An abstract on the results of this work has been submitted for next year's annual conference of the Water Environment Federation. *This grant however does not include support for studying the occurrence of NPEs in local wastewater treatment plants and their river outfalls. Therefore this proposal if funded, will complement the PI's current research and help in establishing a baseline for surfactants in the state of New Jersey.*

Study Design and Experimental Approach

A team consisting of the Principal Investigator, one graduate and one undergraduate student will conduct the proposed research activities. The overall research plan, which is intimately involved with the educational objectives described in the following section, will consist of the following components:

1. *Selection of rivers and wastewater treatment plants representative of high, moderate and low concentration of NPEs.*
2. *Identify sampling points within the wastewater treatment plant to evaluate the physical, chemical and biological removal processes*.
3. *Collect and analyze wastewater and sludge samples at select WWTPs*
4. *Identify sampling points for river water in the major rivers (such as Delaware, Egg Harbor, Raritan) receiving industrial discharges containing APEs.*
5. *Collect and analyze river water and sediments.*

The sample analyses will be conducted at the College of Engineering at Rowan University. Rowan University, founded in 1923 as Glassboro State Teachers College, has evolved into a comprehensive regional state college with six schools including the new College of Engineering. The College of Engineering was initiated using a major gift in 1992 from the Rowan Foundation (Rowan and Smith, 1995). This generous gift has enabled the university to establish perhaps the most innovative and forward-thinking engineering program in the country. The College of Engineering has programs in chemical engineering, civil and environmental engineering, electrical and computer engineering and mechanical engineering. Facilities such as seminar and lecture rooms, laboratories, computer rooms, audiovisual equipment and study hall space are located in

Rowan University's state-of the art $28M Henry M. Rowan Hall. This newly constructed home of the college of engineering has a 92,500 sq. ft. space with multifunctional laboratories. The environmental engineering program has an equipment budget of $358,000 and has all major analytical equipment necessary for this research.

As mentioned earlier, this study will focus on the occurrence of NPEs and their major toxic metabolites such as nonylphenol (NP), nonylphenol monoethoxy carboxylate (NP1EC) and nonylphenol-di and mono-ethoxylates (NP1EO, NP2EO). All three metabolites were shown to be weakly estrogenic to fish, bird and mammal cells in vitro (Giger, 1988). Five major wastewater treatment plants in the North, Central and South Jersey areas and several river reaches representing the average quality of water will be identified for this study. River reaches will be identified with known effluent discharges from industries and municipal wastewater treatment plants. Samples will be collected both during summer (July) and winter (February) as nonylphenol degradation has been shown to be temperature dependent.

Sample Collection

Samples of river water and sediment will be taken along a transect across the mainstream flow at ¼, ½ and ¾ of the distance across the river. Water will be collected from 15 cm below the surface with pre-cleaned 500 mL amber glass bottles. Water samples will also be analyzed for TOC, total dissolved solids (TDS), total suspended solids, temperature, pH, conductivity and dissolved oxygen. Sediment samples will be collected with *a ponar sediment grab sampler*. All samples will be preserved in the field by storing them on ice and later stored at 4^{o}C. In addition 1% (v/v) formalin will be added to all water samples except the ones for TOC for preservation (Naylor, 1992). Twenty-four composite wastewater samples and stabilized sewage sludge from the wastewater treatment plants will also be collected and preserved with formalin. The maximum sample holding time will be 14 days.

Sample Analyses

NP, NP1EO and NP2EO will be extracted from water and sediment/sludge samples using a combined steam distillation/solvent extraction procedure outlined by Ahel and Giger (1985). After addition of 2,4,6-trimethyphenol as an internal standard, the extracts will be analyzed without further purification by normal phase HPLC (Hewlett Packard HPLC 1100). NP1EC will be extracted from acidified water samples with chloroform from separatory funnels. Extracts will be dried by treating with anhydrous sodium sulfate and purified through a silica gel column. The NP1EC will be eluted by methanol and methylated using 1N HCl/methanol and determined by normal phase HPLC (Ahel and Giger, 1985).

Quality Control/Quality Assurance

To ensure raliability of the results, EPA guidelines for quality assurance will be followed throughout the analytical method validation and sample collection and handling. The quality of data collection and overall management of the project will be overseen by the Principal Investigator. All equipment will be calibrated and checked on a routine basis. Standard methods for sampling, sample handling, sample preservation and measurement methods will be followed (Standard Methods for Examining of Water and Wastewater, 19th Edition and Methods for Chemical Analysis of Water and Wastes, USEPA). Analytical grade reagents will be used in all experiments. Students conducting this research will be required to maintain a laboratory notebook that will serve both as a research journal and data acquisition/analysis. All personnel involved in laboratory experiments will be properly trained in correct laboratory procedures and safety guidelines. Experiments will be performed in duplicates or triplicates to make the data statistically sound. Appropriate statistical methods will be used to evaluate the significance/validity of the collected data. Data reduction procedures will be analysis-dependent involving spreadsheet programs (typically Microsoft Excel) and conventional statistical software (SYSTAT, SIGMASTAT).

Impact of the Proposed Project

The findings from these field studies at various wastewater treatment plants and rivers located in New Jersey will have a major impact on the regulatory aspects of APEs. Results of this study will be a significant contribution not only for researchers in New Jersey but all over the U.S. by indicating whether APE degradation products could be problematic or not. The results of this research will be significant in shaping the future of regulations on surfactant production, use and disposal. The wastewater treatment plant community will also benefit tremendously from this study as they are impacted significantly by discharges of nonionic surfactants in their wastewaters. The results of this research will also have a major impact on engineering technology related to in situ bioremediation in the hazardous waste treatment and management field. Engineers must have a thorough understanding of the fate and transport of surfactants for successfully implementing a surfactant based treatment technology. The results of this study will also have a major impact on the disposal of nonionic surfactants by industries involved in manufacturing or utilizing these surfactants in processing plants.

This proposal will also allow the PI to continue quality teaching while focussing on state of the art research activities that are vital to her career development and the graduate program in engineering. Research experiences expose students to the creativity of the research process and enable them to apply their acquired knowledge from formal coursework. Involving undergraduates in research will also encourage them to pursue an advanced degree. This research will also be instrumental in seeking funds and building partnerships with state regulatory agencies, local industries, counties and municipalities.

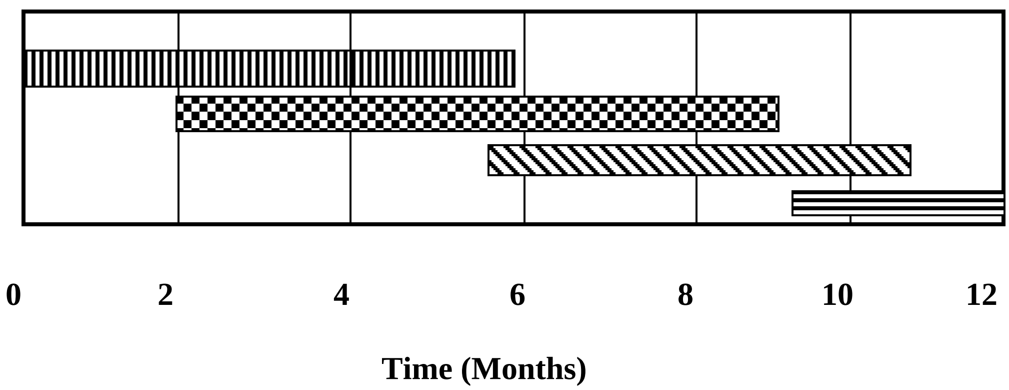
The PI and her co-workers will present their results at professional meetings and conferences to the engineering and scientific communities and regulatory agencies. Students will also be encouraged to participate in local and national poster, presentation and publication competitions arranged by professional organizations. The PI will also be publishing refereed journal papers to disseminate the results of this study.

SCHEDULE OF ACTIVITIES

The major activities for this research are itemized below:

T Survey and Identification of Wastewater Treatment Plants/River Reaches
A Sample Collection for analyses of NPEs and their major intermediates
S Analyses of samples
K Interpretation of Data and Data Analyses, and,
S Preparation of Technical Report and Publications.

A work plan for a year has been presented by means of a Gantt chart below.



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